Viscosity measurement of liquid lithium by the damped torsional oscillation method: experimental study and error analysis

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Abstract

In this paper, a novel oscillating cup viscometer employing the damped torsional oscillation method in conjunction with the Shvidkovskiy calculation methodology was devised for the precise measurement of viscosity in high-temperature liquid lithium. The key factors affecting the experimental results were identified by analysing the propagation of errors in the input parameters of the Shvidkovskiy equation. The results show a very low uncertainty with an average deviation of 1.82% for kinematic viscosity experiments and 1.70% for dynamic viscosity experiments. In addition, a fitting formula within the temperature range is provided, and the dynamic viscosity decreases with increasing temperature. Notably, the experimental deviation using 0.15 mm diameter molybdenum wire was half of that using 0.18 mm diameter wire. Therefore, the paper analysed the influencing factors of liquid lithium viscosity measurement, refined relevant parameters for liquid lithium viscosity, and provided a reference for the testing methods and techniques of the fundamental properties of liquid metals, especially lithium.

Full Text

Preamble

Accurate measurement of viscosity parameters for liquid lithium under high-temperature operating conditions is crucial for thermal-hydraulic modeling of lithium-cooled nuclear reactors. To investigate the mechanisms underlying measurement errors in liquid lithium viscosity, we developed an oscillating cup viscometer based on the Shvidkovskiy algorithm for liquid lithium across a temperature range from 200°C to 650°C, followed by comprehensive experimental error analysis and uncertainty calculations. The results demonstrate remarkably low

uncertainty, with an average deviation of 1.82% for kinematic viscosity measurements and 1.70% for dynamic viscosity measurements. Furthermore, we discovered that using a 0.15 mm diameter molybdenum wire produced experimental deviations nearly half those obtained with a 0.18 mm diameter wire, indicating that appropriately reducing the suspension wire diameter can enhance measurement accuracy in oscillation-based viscosity determinations.

Keywords: Liquid-metal coolant; viscosity; lithium; damped torsional oscillation method; error analysis

1 Introduction

Among alkali metals, liquid lithium is particularly suitable as a coolant for space reactors due to its exceptional properties, including high operating temperature capability, excellent thermal conductivity, and low density [**?**]. During the conceptual design phase of lithium-cooled reactors, the thermophysical properties of liquid lithium across various operating temperatures serve as essential baseline data. Viscosity stands as one of the most critical thermophysical parameters that directly affect coolant flow characteristics and consequently influence reactor system cooling efficiency. However, accurately measuring the viscosity of liquid alkali metals presents significant challenges due to their high melting points, low viscosity values, and high chemical reactivity.

Current viscosity measurement methods include capillary, oscillating vessel, rotational, and oscillating plate techniques [**?**]. For liquid lithium specifically, Andrade [**?**] employed the oscillating sphere method to measure viscosity from near the melting point upward across a certain temperature range. Ban [**?**] measured the viscosity of molten lithium isotopes by observing the damped oscillation of a torsion pendulum. Ito [**?**] utilized an oscillating cup viscometer to measure liquid lithium viscosity in the 464–923 K range, achieving $±$3% accuracy using the Kestin-Newell equation. Similarly, Novikov [**?**], Rigney [**?**], and Achener [**?**] also adopted oscillation methods for their respective viscosity measurements.

Most experimental studies on liquid lithium viscosity date from the mid-to-late 20th century, with deviations among different researchers measuring the same property reaching 15–25%. Achener's results not only show an average deviation of 18% but also exceed 25% deviation compared to other researchers' findings.

In stark contrast to the challenges faced in measuring other liquid metals, Zhu [**?**] investigated an oscillating cup viscometer based on the Shvidkovskiy algorithm with time-resolved reflection measurements for pure aluminum from its melting point to 1300 K, achieving an exceptionally low uncertainty of 2.1% for dynamic viscosity. Patouillet [**?**] successfully measured dynamic viscosity of liquid tin (506–2135 K) and liquid lead (710–1770 K) using an oscillating cup viscometer, maintaining a relatively low uncertainty of approximately 2.5%.

Therefore, to refine measurement methods and parameters for liquid lithium

viscosity, analyze factors contributing to measurement deviations, and elucidate viscosity variation patterns under space environment conditions, this paper presents an experimental study on liquid lithium viscosity using the damped torsional oscillation method. We developed an oscillating-cup viscometer for high-temperature liquid metals and measured liquid lithium viscosity from 200°C to 650°C, revealing the temperature dependence of its kinematic viscosity. Additionally, we analyzed the impact of different molybdenum wire diameters on measurement deviations. Our analysis identifies the oscillation periods T and tAB as the most influential factors, with wire diameter selection directly affecting the suspension system's motion period. The average deviation for kinematic viscosity results is 1.82%, and 1.70% for dynamic viscosity. Both kinematic and dynamic viscosities of liquid lithium decrease with increasing temperature. The dynamic viscosity data deviation is reduced to $±$2.6%, comparable to measurement uncertainties for other liquid metals. This study provides valuable insights and references for advancing measurement methods and techniques for fundamental properties of liquid metals, particularly lithium.

2 Analytical Model for Viscosity of Liquid Lithium

As established, the recognized experimental method for studying liquid metal viscosity is the oscillation method. Considering fabrication difficulties and operational convenience, we selected the hanging-type measurement approach for this research [**?**]. The fundamental principle of the hanging-type oscillation method is illustrated in Fig. 1 [Figure 1: see original paper].

The Shvidkovskiy model, applicable to the oscillation method, was chosen to calculate kinematic viscosity using the following formulas:

$$\nu = \frac{I}{\pi\rho R^4 H p}\left(\frac{1}{T^2}-\frac{1}{T_0^2}\right)\left(1-\frac{2\Delta}{\pi}\right)-\frac{I\delta}{\pi\rho R^4 H p}\left(\frac{1}{T}-\frac{1}{T_0}\right)\left(1-\frac{2\Delta}{\pi}\right)$$

where I is the total moment of inertia, T is the rotation period, δ is the logarithmic decrement, subscript 0 denotes initial parameters, and m stands for the mass of liquid metal. R is the inner diameter of the crucible, H is the height of the liquid sample, and ρ is the liquid density; $p = 1$ indicates partial filling while $p = 2$ indicates complete filling. W is the correction coefficient for different measurement states, and in its calculation, a, b, and c are instrument-related parameters. Estimation methods for these parameters can be found in reference [**?**], where coefficients a, b, and c are functions of y. The calculation sequence for kinematic viscosity is shown in Fig. 2 [Figure 2: see original paper].

Furthermore, we obtain dynamic viscosity as:

$$\eta = \nu\rho$$

The total moment of inertia I is determined through multiple observations of the oscillation period using the following formula:

$$I = \frac{I_i T_0^2 - I_0 T_i^2}{T_i^2 - T_0^2}$$

where I_0 and I_i represent the rotational inertia of the system before and after adding the standard counterweight, respectively. The calculation formulas are:

$$I_0 = \frac{DT_0^2}{4\pi^2}$$

$$I_i = \frac{DT_i^2}{4\pi^2}$$

where m represents the mass of the standard cylindrical load, R_1 and R_2 are its inner and outer radii, respectively, and D is the elastic constant of the molybdenum wire. T_i denotes the time interval of the i-th motion cycle, where each laser spot cycle is divided into four parts: AA, AB, BB, and BA.

Therefore, the calculation formula for T_i is:

$$T_i = t_{AA} + t_{AB} + t_{BB} + t_{BA}$$

The logarithmic decrement δ is measured by determining the time interval between reflections of an oscillating laser beam passing through two laser receivers [**?**]. The calculation formula is:

$$\delta = \frac{1}{2n} \ln \left(\frac{t(AB, i)}{t(AB, i+n)} \right)$$

where $t(AB, i)$ is the AB time interval during the i-th measurement cycle, while $t(AB, i+n)$ denotes the interval during the $(i+n)$-th cycle. T represents the average period of $2n$ consecutive measurements. Over these $2n$ cycles, a set of logarithmic decrement values $(\delta_1, \delta_2, \ldots, \delta_n)$ is obtained, from which the average logarithmic decrement δ is calculated.

The formula for the initial logarithmic decrement δ_0 is:

$$\delta_0 = \frac{c_0}{2\sqrt{kI_0}}$$

where c_0 represents the initial damping coefficient of the suspension wire, k is the stiffness coefficient, and ω_0 denotes the initial angular frequency.

The initial logarithmic decay rate δ_0 can be calculated using:

$$\delta_0 = \frac{1}{2n} \ln \left(\frac{t(AB,0)}{t(AB,n)} \right)$$

Based on Eqs. (1, 2, 4), it can be derived as:

$$\nu = \frac{1}{\pi \rho R^4 H p} \left(\frac{I}{T^2} - \frac{I_0}{T_0^2} \right) \left(1 - \frac{2\Delta}{\pi} \right)$$

The dynamic viscosity is calculated as:

$$\eta = \nu \rho$$

As stated in Eq. (14), inputs for the Shvidkovskiy model to calculate kinematic viscosity include parameters such as the wire elastic coefficient D, the time interval T_i of the i-th motion cycle, the time intervals $t(AB,i)$ and $t(AB,i+n)$, the average cycle T, the idle cycle T_0, the inner and outer radii R_1 and R_2 of the standard cylindrical load, the initial damping coefficient c_0, the stiffness coefficient k, the mass m of the test liquid, the inner diameter R of the crucible, and others. Once these parameters are determined, the final viscosity can be obtained.

3.1 Experimental Setup

The experimental apparatus is a suspended high-temperature molten metal torsional oscillation viscometer designed based on the Shvidkovskiy formula with a correction factor. As illustrated in Fig. 3 [Figure 3: see original paper], it primarily consists of an oscillation system driven by a stepper motor, a heating system, and a measurement system comprising a laser transmitter and receiver.

Oscillation System. As depicted in Fig. 4 [Figure 4: see original paper], the oscillation system is enclosed within the blue box. The crucible, made of stainless steel 316L, has an inner diameter of 13 mm and a height of 40 mm. The metal lithium, after careful impurity removal, is placed in the crucible. The suspension wire is constructed from molybdenum, chosen for its high melting point, high strength at elevated temperatures, and low coefficient of thermal expansion. These properties ensure dimensional stability under high temperatures, facilitating stable and precise logarithmic decay rate measurements and thereby reducing experimental errors.

Heating System. The heating and temperature control system comprises a heating furnace and thermocouple. The crucible rotates in the central chamber of the furnace, with a narrow gap between the crucible's outer wall and heating elements filled with refractory insulation materials to minimize heat loss. The temperature controller employs a Yudian AI-516 intelligent device, capable of precisely regulating the furnace heating rate to achieve specified heating and

cooling curve slopes. This allows rapid attainment of target temperatures with maintained dwell times while compensating for heat loss above the crucible. Testing confirms that the heating system maintains temperature stability within $±$1°C.

Measurement System. Laser oscillation detection is accomplished by the measurement system. The laser source is positioned approximately 1.7 m from a mirror mounted on the suspension system, with laser receivers A and B separated by 55 cm. A helium-neon laser serves as the source, while photodiodes function as photoelectric sensors. Experimental results indicate the system achieves time detection accuracy of 0.1 ms, improving measurement precision for oscillation timing and logarithmic decay. Continuous time interval signals are transmitted to a computer via Bluetooth. The equipment diagram is shown in Fig. 5 [Figure 5: see original paper].

3.2 Lithium Sample Treatment

The experimental crucible measured 13.24 mm in diameter and 40 mm in depth, and the lithium pellets had a purity of 99.974% with 260 ppm total metallic impurities. The small crucible diameter prevented direct impurity removal with a steel spoon. To ensure experimental liquid lithium purity, the lithium grains required pretreatment.

Surface impurities were removed with a knife in an argon glove box, with before-and-after comparisons shown in Fig. 6 [Figure 6: see original paper]. Item No. 1 shows the as-purchased lithium grains with a gray-black surface lacking metallic luster. Melting in this state would make impurity removal difficult within the experimental crucible. Without pretreatment, the liquid lithium would not properly contact and wet the crucible, causing slip between the liquid lithium and crucible inner wall during initial oscillation and resulting in underestimated measurements.

After cutting, the lithium grains were melted in the experimental crucible as shown in Fig. 7 [Figure 7: see original paper]. The crucible was placed in a heating furnace and heated to 400–450°C for approximately 1 hour. Cut lithium grains were placed in the crucible, and the surface tension of liquid lithium was disrupted using a stainless-steel spoon handle to ensure complete wetting of the crucible inner wall. After cooling and solidification, the crucible was sealed using bolts.

4.1 The Effect of Molybdenum Wire Diameter

The oscillation period of the experimental setup is determined by two parameters: the rotational inertia of the reflector, molybdenum rod, and crucible hardware at the suspension system's lower end, and the suspension wire diameter. While moment of inertia is determined by hardware mass and distribution, changing the wire diameter affects the suspension system's rotation period when

the mounted mass is fixed.

Among the input parameters, the inner and outer radii R_1 and R_2 of the standard cylindrical load, the test liquid mass (m), and the crucible inner diameter (R) are independent of wire diameter. Differences in initial damping coefficient (c_0), stiffness coefficient (k), and elastic coefficient (D) between 0.15 mm and 0.18 mm molybdenum wires are negligible. Measurements revealed the elastic constant (D) for 0.15 mm diameter wire to be 1.86×10^{-5} kg · m^2, while for 0.18 mm diameter wire it was 7.73×10^{-5} kg · m^2. Molybdenum wires of both diameters were installed in the suspension system, and standard cylindrical loads (11 mm thick, 1.34 mm inner radius, outer radii of 7.53 mm, 10.02 mm, 12.52 mm, 14.95 mm, and 17.45 mm) were successively mounted. Each experiment was conducted three times.

Experimental results reveal a strong linear relationship between the standard load's rotational inertia ($I - I_0$) and the square of the oscillation period, as shown in Fig. 8 [Figure 8: see original paper]. The curve slope represents the molybdenum wire's elastic coefficient D, indicating that varying standard load masses do not significantly stretch the molybdenum wire. This implies D remains constant and does not affect the oscillation period for loads of identical mass during experiments.

Our measurements show the suspension system's rotational inertia using 0.15 mm diameter molybdenum wire is 3.92×10^{-5} kg · m^2, with an oscillation period T of 9.191 s without a standard load. Using 0.18 mm diameter wire, the measured rotational inertia is 3.90×10^{-5} kg · m^2, and the oscillation period T without a standard load is 4.457 s. Analysis reveals that periods T and t_{AB} are the most significant factors influencing computational results. Furthermore, using different wire diameters directly impacts the suspension system's motion period, causing substantial deviations in viscosity measurement outcomes.

4.2 Uncertainty Calculation

Experimental uncertainty of kinematic viscosity is a crucial parameter for evaluating oscillating cup viscometer performance. Based on experimental data, we determined errors in the Shvidkovskiy equation's input parameters and analyzed their propagation during kinematic viscosity calculations. The final result accuracy is controlled by these errors, with each parameter's measurement precision presented in Table 1 .

In the Shvidkovskiy model, input parameters for calculating kinematic viscosity include the suspension system's rotational inertia, empty cup oscillation period and logarithmic decay rate, sample-loaded oscillation period and logarithmic decay rate, sample density and mass, and crucible inner radius. To analyze experimental uncertainty, we determined root mean square deviations for these parameters. The measured period's relative error was less than 0.04%, which is negligible. The uncertainty of δ was 0.3%. Similarly, uncertainties for the empty cup's oscillation period and logarithmic decay rate were 0.006% and

0.2%, respectively. Reasonable estimates were made for other input parameters' uncertainties. Through repeated mass measurements, mass uncertainty was approximately 0.4%. The sample crucible's inner radius uncertainty was 0.1%. Considering thermal expansion and material changes during heating, $\Delta R/R$ was estimated at 0.3%. The suspension system's rotational inertia I uncertainty was approximately 0.5%. Based on these values, the total estimated dynamic viscosity uncertainty was calculated as:

$$S_\nu = \sqrt{\sum_{i=1}^{n} \left(\frac{\partial \nu}{\partial x_i} S_i \right)^2}$$

where S_ν represents kinematic viscosity uncertainty and S_i denotes uncertainty for each measured parameter. Consequently, the total experimental error for the oscillating cup viscometer is estimated at approximately 1.8%. Table 2 lists uncertainties for various measurement parameters.

5 Results and Discussion

Multiple experimental sets were conducted using 0.15 mm and 0.18 mm molybdenum wires. Time-period data were obtained for temperatures ranging from 200°C to 650°C at 50°C intervals under different suspension wires. Kinematic and dynamic viscosities were then calculated for different wire diameters and temperatures using the derived formulas. Three valid experimental result sets were obtained, with one shown in Table 3 and remaining data displayed within error bars in the figures.

5.1 Kinematic Viscosity of Liquid Lithium

Using literature data as standard values [**?**], measured lithium densities obtained with different molybdenum wire diameters were compared to standard values at various temperatures. The overall deviation is illustrated in Fig. 9 [Figure 9: see original paper], where ν_{exp} represents experimental values and ν_{eq} represents reference values. A comparison between experimental and reference values appears in Fig. 10 [Figure 10: see original paper]. Using our experimental setup, the minimum kinematic viscosity measurement deviation was 0.507%, the maximum was -3.34%, and the average was 1.82%. Comparing wire diameters, the 0.15 mm molybdenum wire exhibited a minimum deviation of -0.33% and maximum deviation of $±$1.72%, while the 0.18 mm wire showed a minimum deviation of 1.25% and maximum deviation of $±$3.3%.

Because the 0.18 mm molybdenum wire's elastic modulus is 4.16 times that of the 0.15 mm wire, the corresponding vibration period differs by 2.06 times during empty measurements. Under identical moment of inertia, the elastic constant is proportional to the period squared. In logarithmic decay rate calculations, 0.18 mm and 0.15 mm wires were employed respectively. For the 0.18 mm wire, the time interval between $t(AB, i)$ and $t(AB, i + n)$ from vibration

start to end was approximately 1.3 s, averaging 90 measured cycles. For the 0.15 mm wire, the $t(AB,i)-t(AB,i+n)$ interval was about 2.5 s, yielding an average of 170 measured cycles. The longer time interval and higher cycle count indicate the suspension system's vibration more closely approximates actual resistance decay. Under 1 ms time resolution, a 1 ms deviation in measuring $t(AB,i+n)$ contributed a 3% impact on final kinematic viscosity calculation for the 0.18 mm wire, while a 1 ms deviation in measuring $t(AB,i)$ led to 7–8% impact. In contrast, for the 0.15 mm wire, a 10 ms deviation in measuring $t(AB,i+n)$ had a 2.4% impact, and a 1 ms deviation in measuring $t(AB,i)$ had a 3.5% impact. Statistical analysis of all measured cycles revealed that for identical wire diameters and loading conditions, time intervals between $t(AB,i)$ and $t(AB,i+n)$ remained constant for the first 10 cycles, with fluctuations occurring only in subsequent cycles. Therefore, the primary influence on calculation results stemmed from measurement deviation at point $t(AB,i+n)$. As evident from Fig. 9's deviation results, experiments using the 0.15 mm molybdenum wire with 1 ms time resolution yield more stable and accurate results.

5.2 Dynamic Viscosity of Liquid Lithium

Experimentally measured kinematic viscosities were calculated and compared using both literature reference values and experimental values. A comparative analysis of dynamic viscosity data deviations appears in Fig. 11 [Figure 11: see original paper], where ρ_{exp} represents experimental values and ρ_{eq} represents literature reference values. Direct comparison between experimental and literature reference values is depicted in Fig. 12 [Figure 12: see original paper].

As observed in Fig. 12, the maximum dynamic viscosity deviation compared to reference literature values is 3.0%, with a minimum deviation of 0.58% and an average deviation of 1.70%. Notably, deviation fluctuations are reduced after calculating dynamic viscosity from kinematic viscosity data obtained using the 0.18 mm diameter molybdenum wire, resulting in an average deviation of \$±\$2.6%. No significant differences were observed in dynamic viscosity data calculated using different density values.

6 Conclusion

This paper presents the development of an oscillating cup viscometer using the Shvidkovskiy algorithm for liquid lithium across 200°C to 650°C, along with measurement error analysis and uncertainty evaluation. Key findings include:

1. The average deviations for liquid lithium's kinematic and dynamic viscosities in the 200°C to 650°C range are 1.82% and 1.70%, respectively, demonstrating acceptable measurement accuracy for the developed viscometer.

2. Motion periods are significant input parameters for the Shvidkovskiy model, and wire diameter is a critical factor that can cause apparent

viscosity measurement deviations by impacting the suspension system's motion period.

3. Experimental results show that the maximum kinematic viscosity deviation using 0.15 mm molybdenum wire is $±$1.72%, superior to the $±$3.54% deviation with 0.18 mm wire, indicating that smaller wire diameters provide better resistance to uncertainty perturbations.

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Note: Figure translations are in progress. See original paper for figures.